

1021050



# PATENT SPECIFICATION

DRAWINGS ATTACHED

1021,050

Date of Application and filing Complete Specification: June 4, 1963.  
No. 22178/63.

Application made in United States of America (No. 201,145) on June 8, 1962.  
Complete Specification Published: Feb. 23, 1966.  
© Crown Copyright 1966.

Index at acceptance:—B1 B(3A, 5N)

Int. Cl.:—B 01 d

## COMPLETE SPECIFICATION

### Apparatus for use in Separating Gases from Liquids

We, HALCON INTERNATIONAL, INC., of 2, Park Avenue, New York 16, New York, United States of America, a corporation organized and existing under the laws of the

5 State of Delaware, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to apparatus for use in the separation of mixtures of gases and liquids under elevated pressure and temperature conditions.

15 High pressure separators are used in many commercial processes and they may be made up in several known ways. The vessel may be in the form of a vertical cylinder having very thick walls. The gas-liquid mixture may be

20 fed in near or at the top thereof, and after separation, the gas may be removed via an appropriate valve opening near the top, and the liquid may be removed via an appropriate opening near or at the bottom thereof. One

25 means of determining the liquid level therein is by so called radiation type liquid level indicators which involve passing radiation from one side of the vessel to the other and detecting the change in radiation passed therethrough

30 with suitable means such as a Geiger counter when the path changes from gas to liquid. During operation, the vessel may be allowed to accumulate liquid, i.e. the liquid may be drawn-off intermittently or continuously.

35 The liquid mixture to be separated may require maintenance under high pressure and temperature conditions, and may, for example, be an ethylene polymerization reaction mixture containing gaseous ethylene as well as

40 liquid polymer which is separated at a pressure of about 2,500 to 15,000 p.s.i.g., more usually 4,000 to 15,000 p.s.i.g. and a temperature of about 200° to 575° F. The vessel then has to be provided with means for maintaining this temperature; i.e. to prevent radiant

heat loss, and steam jacketing has heretofore been proposed.

50 However, the steam jacketing means heretofore proposed have been welded directly to the outer wall of the vessel and have required complicated bellows expansion means. In addition, the radiation device ports were covered by welded caps. The art is confronted by the problem of providing efficient jacketing means which eliminates the completed bellows requirements as well as providing ready access to and accurate determination of the location of the radiation device ports.

55 According to the invention apparatus for phase-separating a gas-liquid mixture at an elevated temperature and pressure comprises a thick-walled vessel provided with a gas liquid feed inlet, a gas outlet at or near the top thereof, a liquid outlet at or near the base thereof, and a plurality of pairs of horizontal ports extending from the outside into but not through the wall of the vessel, the two ports of each pair having a common horizontal axis passing through the inside of the vessel and the pairs of ports being at different horizontal levels in the vessel, and a heating jacket surrounding and in contact with the outer surface of the vessel but free to move relative thereto under the influence of temperature differences between the vessel outer surface and the jacket, the jacket being provided with heating fluid inlet and outlet and with a plurality of passages through the jacket but sealed from the inside thereof, each passage being in line with and communicating with one of said ports.

60 65 70 75 80 85 90  
The outer wall of the vessel may advantageously be cylindrical in shape and the surrounding steam jacket annular in shape, one end of the jacket being attached to the vessel wall. The annular jacket may be made up of an inner wall and an outer wall with an annular spacer at each end of the jacket and in such a case each of the walls is attached to the spacer at each end.

[Price 4s. 6d.]

Each of the passages through the heating jacket may comprise a sleeve passing through an opening in the outer wall and set on the inner wall surrounding an opening through the inner wall which is smaller than the outer wall opening, the inner opening being large enough to permit access to the contiguous or facing port in the vessel and the sleeve being attached to each of the walls at its contact points therewith.

The apparatus of the invention may comprise a vessel built-up of layers and provided with at least one tell-tale hole for the detection of leakage in the laminations, the jacket being provided with a passage communicating with each of the tell-tale holes.

One embodiment of the invention is illustrated in the accompanying drawing, in which:

Figure 1 is a cross sectional view of a separator provided with a steam jacket in accordance with the invention; and

Figures 2 and 3 are enlarged fragmentary sections showing the arrangement of the ports and the access passages thereto.

Referring to the drawing, the high pressure separator is constituted by a vessel 10 having a thick steel wall 11 and made by forging, laminating, or in any other convenient manner. The vessel 10 is provided with a liner 12 of corrosion resistant material such as stainless steel. The vessel 10 is also provided with a heating fluid jacket 13 formed by an inner wall 14, in contact with the outer surface of the wall 11, and an outer wall 15. The walls 14 and 15 are spaced and sealed by members or spacers 16 to which each wall is welded. There is one of such spacers at the bottom and also one at the top of the jacket. The inner jacket wall 14 is in contact with the vessel wall 11 but free to move relative thereto. The upper part of the jacket 13 is attached to the steel wall 11 via weld 17. The jacket 13 is provided with a series of passages 18 arranged to provide access to cavities or ports 20 and 20<sup>1</sup> drilled part way into the wall 11. The ports 20 are for introducing radiation from a usual radioactive source such as cobalt 60 and the ports 20<sup>1</sup> are for passing out and detecting the radiation.

The vessel 10 is provided with inlet means 21 for introducing the mixture of gas and liquid, gas outlet means 22, relief valve means 23, guide or baffle means 24 and also guide or baffle means 24<sup>a</sup>, and liquid outlet means 25. The jacket 13 is provided with fluid heating medium inlet means 26 and outlet means 27. All of said means are provided with appropriate valves (not shown).

Steam is a preferred heating fluid but any other suitable fluid may be used.

If the vessel 10 is built-up from a series of layers, one or more tell-tale holes 28 are provided in usual manner, and a jacket passage 18<sup>1</sup> is provided for each.

Referring to Figure 2, the access passage in the steam jacket 13 for each port 20 includes a tubular member 19 welded by welds 19a to the inner wall 14 and the outer wall 15 of the jacket. The access passages for the ports 21 are similarly constructed as shown in Figure 3.

During start-up of a plant including this separator, such as a plant for making polyethylene from ethylene, the separator is brought up to temperature by passing steam through the jacket. This is accomplished efficiently in accordance with the invention without any buckling or the like problems, even through the bellows means of prior suggestions is eliminated. The tube walls of the annular steam jacket expand together and the inner wall is free to move lengthwise relative to the wall of the vessel without restraint.

The present apparatus completely avoids the problem of welding caps or covers for the radioactive radiation means, which may be set near or in ports 20. This facilitates location and use of automatic or semi-automatic means for introducing or retracting the radiation source into or from the bottom of the port.

The reaction mixture of ethylene and polyethylene prepared in known manner may be separated in the vessel at a pressure of about 2,500 to 15,000 p.s.i.g. and a temperature of about 200° to 575° F. This reaction mixture may be prepared in accordance with the reaction conditions set forth in British Patent specification No. 816,110. The liquid polymer is allowed to build-up to a level of one of the pairs of ports above the lowest pair of ports and then the valve may be opened until the level recedes to that of a lower pair of ports at which time the valve is closed and the procedure is repeated. If the rate of feed to the vessel is appropriately regulated, the liquid draw-off may be continuous, the level of the liquid in the vessel being maintained above that of the lowest pair of ports but below one of the other pairs of ports. Two or more pairs of ports may be used as desired, three being shown in Figure 1 above the lowest pair.

Although a cylindrical type vessel is preferred since it may be provided with an annular jacket other shapes may be used if desired, providing the inner jacket wall is substantially continuously in contact with the outer wall of the separator vessel and the jacket has freedom of relative movement which may be caused by temperature differences.

#### WHAT WE CLAIM IS:—

1. Apparatus for phase-separating a gas-liquid mixture at an elevated temperature and pressure which comprises a thick-walled vessel provided with a gas/liquid feed inlet, a gas outlet at or near the top thereof, a liquid outlet at or near the base thereof, and a plural-

70

75

80

85

90

95

100

105

110

115

120

125

130

- ity of pairs of horizontal ports extending from the outside into but not through the wall of the vessel, the two ports of each pair having a common horizontal axis passing through the inside of the vessel and the pairs of ports being at different horizontal levels in the vessel, and a heating jacket surrounding and in contact with the outer surface of the vessel but free to move relative thereto under the influence of temperature differences between the vessel outer surface and the jacket, the jacket being provided with heating fluid inlet and outlet and with a plurality of passages through the jacket but sealed from the inside thereof, each passage being in line with and communicating with one of said ports.
2. Apparatus as claimed in claim 1 wherein the outer wall of the vessel is cylindrical in shape and the jacket is annular in shape, one end of the jacket being attached to the vessel wall.
3. Apparatus as claimed in claim 2 wherein the jacket is made up of an inner wall and an outer wall, each end of the jacket is provided with an annular spacer and each of the walls is attached to the spacer at each end.
4. Apparatus as claimed in claim 1, 2 or 3 wherein each of the jacket passages comprises a sleeve passing through an opening in the outer wall and set on the inner wall surrounding an opening through the inner wall which is smaller than the outer wall opening, the inner wall opening being large enough to permit access to the contiguous port in the vessel and the sleeve being attached to each of the walls at its contact points therewith.
5. Apparatus as claimed in any of claims 1 to 4 wherein the vessel is built-up of layers and is provided with at least one tell-tale hole, the jacket being provided with a passage communicating with each of the tell-tale holes.
6. Apparatus for use in separating a mixture of gas and liquid at elevated temperature and pressure, constructed substantially as described herein with reference to the accompanying drawing.

MATHYS & SQUIRE,  
Chartered Patent Agents,  
Staple House, 51/52, Chancery Lane,  
London, W.C.2.  
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1966. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

1021050

COMPLETE SPECIFICATION

1 SHEET

*This drawing is a reproduction of  
the Original on a reduced scale*

